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Structure of 5-Amino-1-(2,6-dimethylphenyl)-3-methylthio-1*H*-1,2,4-triazole

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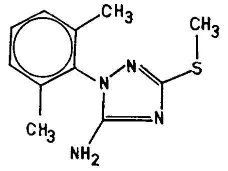
Abstract. $C_{11}H_{14}N_4S$, $M_r = 234{\cdot}33$, monoclinic, $P2_1/c$, $a = 15{\cdot}707$ (3), $b = 7{\cdot}748$ (4), $c = 20{\cdot}519$ (8) Å, $\beta = 98{\cdot}61$ (2)°, $V = 2469$ (2) Å^3, $Z = 8$, $D_x = 1{\cdot}260$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}20$ mm^{-1}, $F(000) = 992$, $T = 293$ (2) K, $R = 0{\cdot}055$ for 3940 observed reflections. The conformation of the two symmetry-independent molecules (conformers) significantly differs in the orientations of the phenyl rings and *S*-methyl moieties with respect to the triazole rings. The features of the planar *s*-triazole rings are governed by the rules reported by Kálmán & Argay [*J. Mol. Struct.* (1983), **102**, 391–402]. Of the four amino H atoms of the two conformers, three take part in hydrogen bonds with the N-atom lone pairs.

Introduction. The title compound was prepared by Reiter, Somorai, Jerkovich & Dvortsák (1982). The colourless crystals melt at 401–403 K. The molecular structure (1) inferred from spectroscopic data was substantiated by X-ray diffraction. Measurements of temperature dependence of electric permittivity (Koczó, Dulić & Horvát, 1984) show a rapid change of permittivity at about 368 K and indicate a phase transformation. To explain the phase-transformation mechanism it is necessary to know the structure at room temperature.



(1)

Experimental. Crystal *ca* 0·20 × 0·36 × 0·50 mm. Philips PW 1100 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $0{\cdot}049 \le (\sin\theta)/\lambda \le 0{\cdot}707$ Å^{-1}, θ–2θ scan, h −21 to 19, k 0 to 10, l 0 to 28. Cell parameters by least squares using 20 centred reflections with θ range from 6·03 to 9·50°. Systematic absences: $l = 2n+1$ in $h0l$ and $k = 2n+1$ in $0k0$. Of 4348 unique reflections, 3940 with $I > 2{\cdot}5\sigma(I)$ were taken as observed. No absorption correction was performed. Three standard reflections (600; $\bar{1}\bar{1}6$; $0\bar{1}6$) were monitored every 2 h. Max. and min. corrections of decay were 1·00 and 0·97. Structure was solved by *SHELX*76 (Sheldrick, 1976) and successive structure-factor, Fourier and difference-Fourier calculations. Full-matrix refinement. $w(\Delta F)^2$ was minimized for 32 non-H atoms (292 parameters). Final $R = 0{\cdot}055$, $wR = 0{\cdot}062$, $w = 2{\cdot}5275[\sigma^2(F) + 15{\cdot}31 \times 10^{-4}F^2]^{-1}$. Max. and min. peak heights in final $\Delta\rho$ map +0·33 and −0·28 e Å^{-3}. Positions of H atoms were generated from assumed geometries with C—H = 1·08 Å, the remaining H atoms bound to N atoms were located in a difference Fourier map. They were included in the final structure-factor calculations with individual isotropic temperature factors. Max. $\Delta/\sigma = 0{\cdot}92$. Scattering factors as in *SHELX*76. Calculation on IBM 43/41 computer.*

Discussion. Atomic coordinates of non-H atoms are in Table 1. The molecular geometry with standard atomic numbering for both symmetry-independent molecules (I) and (II) is shown in Figs. 1 and 2. The bond lengths and angles for non-H atoms are listed in Table 2. The conformation of the symmetry-independent molecules differs in the rotation of the dimethylphenyl rings and the *S*-methyl moieties about the N1—C7 and S—C3

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43925 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Final fractional coordinates for non-*H *atoms, with e.s.d.'s in parentheses*

$$U_{\rm eq} = \tfrac{1}{3}\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$(Å^2)
S	1·0021 (1)	0·2289 (1)	0·1857 (1)	0·0494 (4)
N1	0·8281 (1)	−0·0419 (3)	0·0835 (1)	0·043 (1)
N2	0·8570 (1)	0·0427 (3)	0·1429 (1)	0·044 (1)
C3	0·9311 (2)	0·1053 (3)	0·1312 (1)	0·037 (1)
N4	0·9531 (1)	0·0688 (3)	0·0712 (1)	0·038 (1)
C5	0·8866 (1)	−0·0238 (3)	0·0425 (1)	0·036 (1)
N6	0·8773 (1)	−0·0892 (3)	−0·0188 (1)	0·048 (1)
C7	0·7511 (2)	−0·1421 (4)	0·0745 (1)	0·041 (1)
C8	0·7543 (2)	−0·3095 (4)	0·0995 (1)	0·049 (1)
C9	0·6781 (3)	−0·4044 (5)	0·0909 (2)	0·067 (1)
C10	0·6032 (2)	−0·3329 (5)	0·0570 (2)	0·072 (2)
C11	0·6023 (2)	−0·1707 (5)	0·0317 (2)	0·066 (1)
C12	0·6760 (2)	−0·0699 (4)	0·0402 (2)	0·050 (1)
C13	0·6753 (3)	0·1098 (5)	0·0121 (2)	0·075 (2)
C14	0·8364 (2)	−0·3864 (5)	0·1349 (2)	0·069 (1)
C15	0·9479 (2)	0·2324 (5)	0·2560 (2)	0·063 (1)
S′	0·8503 (1)	0·0800 (1)	0·4683 (1)	0·0588 (4)
N1′	0·7076 (1)	−0·1996 (3)	0·3457 (1)	0·040 (1)
N2′	0·7427 (1)	−0·1568 (3)	0·4103 (1)	0·044 (1)
C3′	0·7928 (2)	−0·0271 (3)	0·4005 (1)	0·041 (1)
N4′	0·7957 (1)	0·0164 (3)	0·3370 (1)	0·043 (1)
C5′	0·7416 (2)	−0·0948 (3)	0·3043 (1)	0·039 (1)
N6′	0·7197 (2)	−0·0988 (4)	0·2371 (1)	0·058 (1)
C7′	0·6466 (2)	−0·3375 (3)	0·3336 (1)	0·039 (1)
C8′	0·6750 (2)	−0·4986 (4)	0·3169 (1)	0·049 (1)
C9′	0·6146 (2)	−0·6320 (4)	0·3079 (2)	0·066 (2)
C10′	0·5313 (3)	−0·6027 (5)	0·3157 (2)	0·073 (2)
C11′	0·5040 (2)	−0·4426 (5)	0·3326 (2)	0·068 (2)
C12′	0·5614 (2)	−0·3049 (4)	0·3417 (2)	0·051 (1)
C13′	0·5347 (2)	−0·1274 (5)	0·3612 (2)	0·075 (2)
C14′	0·7685 (2)	−0·5304 (5)	0·3106 (2)	0·071 (2)
C15′	0·9423 (2)	0·1507 (5)	0·4326 (2)	0·068 (1)

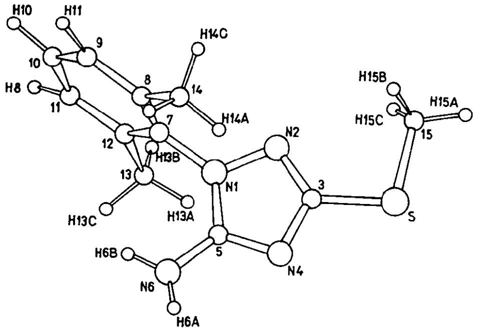

Fig. 1. A perspective view of molecule (I) showing atomic numbering.

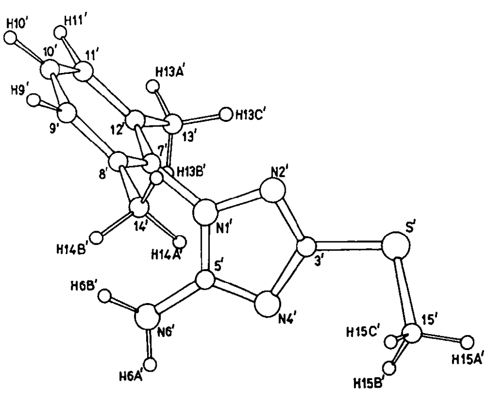

Fig. 2. A perspective view of molecule (II) showing atomic numbering.

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

	(I)	(II)		(I)	(II)
S—C3	1·742 (3)	1·751 (3)	C7—C8	1·392 (4)	1·386 (4)
S—C15	1·781 (3)	1·801 (3)	C8—C9	1·393 (4)	1·397 (4)
N1—N2	1·397 (3)	1·396 (3)	C9—C10	1·390 (5)	1·361 (5)
N1—C5	1·343 (3)	1·342 (3)	C10—C11	1·359 (5)	1·374 (5)
N1—C7	1·425 (3)	1·432 (3)	C11—C12	1·386 (4)	1·392 (4)
N2—C3	1·317 (3)	1·310 (3)	C12—C7	1·397 (4)	1·395 (4)
C3—N4	1·357 (3)	1·354 (3)	C12—C13	1·506 (5)	1·510 (4)
N4—C5	1·331 (3)	1·321 (3)	C8—C14	1·506 (4)	1·513 (4)
C5—N6	1·344 (3)	1·371 (4)			
C3—S—C15	101·0 (1)	99·9 (1)	N1—C7—C8	118·5 (2)	118·9 (2)
S—C3—N2	125·7 (2)	119·5 (2)	N1—C7—C12	118·9 (2)	118·2 (2)
S—C3—N4	118·1 (2)	124·0 (2)	C8—C7—C12	122·7 (2)	122·9 (3)
N1—N2—C3	101·2 (2)	101·4 (2)	C7—C8—C9	117·5 (3)	117·5 (3)
N2—C3—N4	116·2 (2)	116·5 (2)	C7—C8—C14	121·8 (3)	121·4 (3)
C3—N4—C5	102·9 (2)	102·3 (2)	C9—C8—C14	120·7 (3)	121·1 (3)
N4—C5—N1	110·2 (2)	111·1 (2)	C8—C9—C10	120·0 (3)	120·5 (3)
N4—C5—N6	125·8 (2)	124·8 (2)	C9—C10—C11	121·3 (3)	121·5 (3)
N1—C5—N6	124·0 (2)	124·1 (2)	C10—C11—C12	120·8 (3)	120·3 (3)
C5—N1—N2	109·5 (2)	108·6 (2)	C11—C12—C7	117·7 (3)	117·3 (3)
C5—N1—C7	128·4 (2)	131·3 (2)	C11—C12—C13	121·0 (3)	122·2 (3)
N2—N1—C7	121·8 (2)	120·0 (2)	C7—C12—C13	121·4 (3)	120·4 (3)

Table 3. *Intermolecular hydrogen bonds*

		N···N (Å)	H···N (Å)	∠NH···N (°)
N6—H*A*···N4	(2−x, −y, −z)	3·022 (3)	2·20 (3)	164 (1)
N6—H*B*···N2′	(x, −y−$\frac{1}{2}$, z−$\frac{1}{2}$)	3·088 (3)	2·19 (3)	164 (1)
N6′—H*A*′···N2	(x, y, z)	3·292 (4)	2·35 (3)	173 (1)

bonds. The differences can be expressed by the torsion angles: N2—N1—C7—C8 −80·4 (4), N2′—N1′—C7′—C8′ −98·4 (4), C15—S—C3—N2 2·2 (3) and C15′—S′—C3′—N2′ 150·8 (3)°. In both molecules the sequence of the endocyclic bond-angle magnitudes (°) (given by their mean values) of the planar *s*-triazole

$$\begin{array}{ccccccccc} \mathrm{N1} & & \ddot{\mathrm{N}}2 & & \mathrm{C3} & & \ddot{\mathrm{N}}4 & & \mathrm{C5} \\ 109 & > & 101 & \ll & 116 & \gg & 103 & < & 111 \end{array}$$

rings are governed by the rules reported by Kálmán & Argay (1983). Of the four amino H atoms, three are involved in hydrogen bonds with the lone pairs of the N2, N2′ and N4 atoms (Table 3) forming a three-dimensional network.

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